IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Re: Application of Jacobsen et al.

Serial No: 10/523,828

Filed: 02/08/2005

For: SUPPORTED POLYMERISATION CATALYSTS

DECLARATION OF GRANT BERENT JACOBSEN

I, Grant B. Jacobsen, do hereby solemnly declare as follows:-

1. I am a [nationality] subject, residing at [address].

2. I am familiar with the technical details concerning the US Patent Application Serial No. 10/523,828 filed on 8th February 2005 of which I am an inventor.

3. I am also familiar with the technical details concerning the US Patents 5,783,512 and 6,271,165, hereinafter D1 and D2 respectively, on both of which I am also an inventor.

4. In the Office Action dated September 27th 2006, the Examiner has stated that the claims of the present invention are obvious over both D1 and D2.

5. Both D1 and D2 disclose methods for formation of supported transition metal catalysts and processes for using the catalysts for polymerisation of ethylene.

6. The general disclosure of both D1 and D2 is however that the support is optional and that the order of the addition of the specific components is not critical (e.g. D1, col. 17, lines 28-35; D2, col. 26, lines 58-67. The documents also both describe a large number of different organometal compounds that may be used, of which alkylaluminium compounds are preferred (e.g. D1, col. 13, line 25 onwards; D2, col. 21, line 56 onwards).

7. In contrast the method of the present invention requires specific use of an aluminoxane rather than any alternative organometal compounds, specific use of a support, and a specific order of addition of the components wherein the aluminoxane and the ionic activator are mixed together and added to the support prior to addition of a transition metal compound.

8. The present invention, and in particular the use of aluminoxanes, has been found to result in an advantageous activity profile for the resultant catalysts (see p.2, 6th paragraph of the application as filed). In the Examples, this is represented by a much lower reaction exotherm than conventionally observed. The invention has also been found to give a polymer with high

melt strength (5.9cN in the polymer from Example 2 – see p.12, last line as filed).

9. The Examiner notes a number of Examples from D1 and D2. However, none of these Examples exemplify the use of aluminoxane. A number of the Examples, e.g. Examples 6 of both D1 and D2, do however exemplify an equivalent method of the present invention but using an alkylaluminium (TEA) as the organometal.

10. The attached Appendix 1 presents data from an equivalent process to these Examples of D1 and D2. These results show not only that the initial exotherm from the use of an aluminoxane compared to an alkylaluminium is much lower (shown as absence of a sharp peak in the activity), but that the resulting polymers have significantly increased melt strength.

11. These advantages of the process of the present invention are not obvious from either D1 or D2.

All statements made of my own knowledge are true and all statements made on information and belief are believed to be true. I also understand that willful false statements and the like are punishable by fine or imprisonment, or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon.

Signed:

GRANT BERENT JACOBSEN

Date: 12 DECEMBER 2006

Appendix 1

Comparative catalyst preparation

(i) Treatment of silica

A suspension of Grace 948 silica (13kg, previously calcined at 250°C for 5 hours) in 110 litres (L) of hexane was made up in a 240L vessel under nitrogen. 1L of a hexane solution containing 2g/L of Stadis 425 was added and stirred at room temperature for 5 minutes. 29.1L of a 892mmolAl/L solution of triethylaluminium (TEA) in hexane was added slowly to the stirred suspension over 30 minutes, while maintaining the temperature of the suspension at 30°C. The suspension was stirred for a further 2 hours. The hexane was filtered, and the silica washed with hexane, so that the aluminium content in the final washing was less than 0.5 mmol Al/litre. Finally the suspension was dried *in vacuo* at 60°C to give a free flowing treated silica powder with residual solvent less than 0.5wt%.

(ii) Catalyst fabrication

All steps, unless otherwise stated, of the catalyst fabrication were carried out at 20°C. 3L of toluene was added to a 24L vessel equipped with a turbine stirrer, and stirred at 300rpm. 5.01L of a 9.5wt% solution in toluene of bis(hydrogenated tallow alkyl) methyl ammonium tris(pentafluorophenyl)(4-hydroxyphenyl)borate was added during 15 minutes. Then 1.57L of a 250mmolAl/L solution in toluene of triethylaluminium was added during 15 minutes and mixture stirred for 30 minutes. The solution obtained was then transferred under nitrogen, with stirring during 2 hours, to an 80L vessel containing 10kg of the TEA treated silica described above. 60L of hexane was then rapidly introduced and mixed for 30 minutes. 1.83 kg of a 7.15wt% solution in heptane of (t-butylamido)(tetramethyl-η^5-cyclopentadienyl) dimethylsilanetitanium -η^4-1,3-pentadiene was added during 15 minutes. Mixing was continued for 1 hour and 1L of a 2g/L hexane solution of stadis 425 was added. The catalyst slurry was then transferred to a vessel of volume 240L and 70L of hexane added. Excess solvent was removed by decantation, and a further 130L of hexane added. This process was repeated until less than 0.2L of toluene remained in the solvent. 1L of a 2g/L hexane solution of stadis 425 was then added and the catalyst dried under vacuum at 40°C to a residual solvent level of 1wt%.

This example is comparable to Examples 6 of both US 5,783,512 and US 6,271,165, and uses triethylaluminium (TEA) instead of aluminoxane as required by the present invention.

Experimental

The comparative catalyst produced above was tested under similar conditions to Example 3 of the present application. The reaction profile for this is shown as A in Figure 1 below and compared to Example 3 using the catalyst of Example 2 according to the present application, which is shown as B.

The increase in activity profile shown initially is directly related to the reaction exotherm. It can be seen that the exotherm with the Example according to the present invention (B) is significantly lower than that observed for the equivalent reaction using the comparative catalyst (A), i.e. using a catalyst comparable to those of D1 and D2 formed using an alkylaluminium instead of an aluminoxane. This would be expected to result in an improvement of the operability of the catalyst at industrial scale.

Figure 2 shows the melt strength of the relevant polymers produced in the comparison. Again a marked improvement (increase) is shown with the Example according to the present invention (B) compared to the results observed for the equivalent reaction using the comparative catalyst (A), i.e. using a catalyst comparable to those of D1 and D2 formed using an alkylaluminium instead of an aluminoxane.